

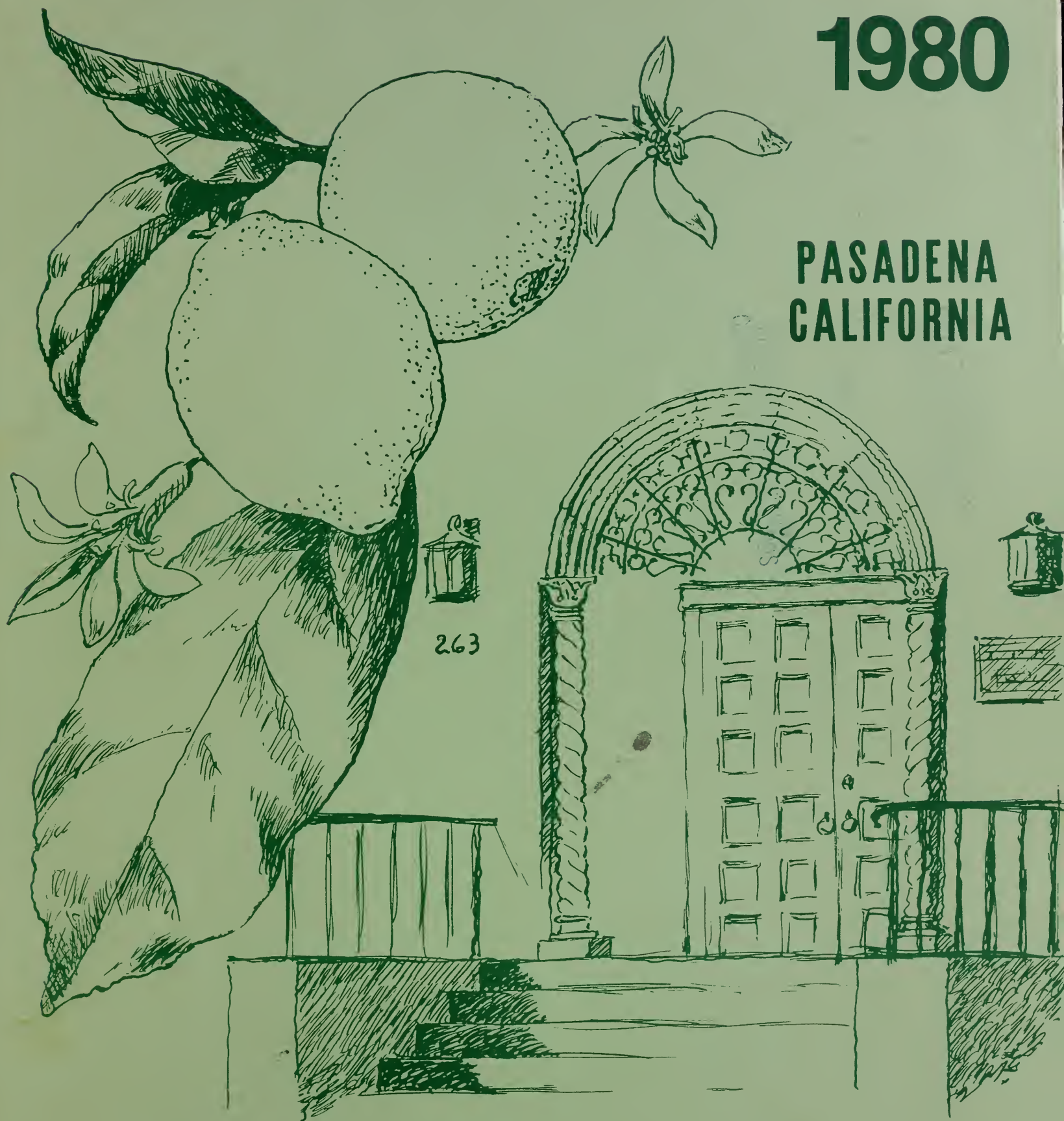
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PASADENA
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CITRUS RESEARCH CONFERENCE

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CITRUS RESEARCH CONFERENCE

December 11, 1980

Pasadena, California

ABSTRACTS OF PAPERS

Sponsored By:

Fruit and Vegetable Chemistry Laboratory
263 South Chester Avenue
Pasadena, California 91106

Agricultural Research
Science and Education Administration
UNITED STATES DEPARTMENT OF AGRICULTURE

FOREWORD

This Citrus Research Conference is being held to bring to members of the citrus and allied industries the latest results of research on the chemistry, biochemistry, and technology of citrus fruits and their products carried on by Agricultural Research, Science and Education Administration, U.S. Department of Agriculture. The following are participating in or contributing to this year's conference:

Western Region

Fruit and Vegetable Chemistry Laboratory
263 South Chester Avenue, Pasadena, California 91106

Western Regional Research Center
800 Buchanan Street, Albany, California 94710

Southern Region

U.S. Citrus and Subtropical Products Laboratory
600 Avenue S, N.W., Winter Haven, Florida 33880

Food Crops Utilization Research Laboratory
Weslaco, Texas 78596

U.S. Horticultural Research Laboratory
2120 Camden Road, Orlando, Florida 32803

Conference Headquarters:

Huntington-Sheraton Hotel
1401 South Oak Knoll Avenue
Pasadena, California 91109

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Thursday, December 11, 1980

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California-Hawaii Area
USDA-SEA-AR, Fresno, California

INTRODUCTORY REMARKS: C. Gordon Beisel, Manager
Products Research and Development Division
Sunkist Growers, Inc.
Ontario, California

CHAIRMAN: Vincent P. Maier, Research Leader
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

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PROGRESS IN ISOLATING A HESPERIDIN-SOLUBILIZING
FACTOR FROM LEMON LEAVES*

Raymond D. Bennett and Ronald E. Schuster
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

V200

Previous work had shown that lemon leaves contain material of relatively low molecular weight ($\leq 1,000$) which solubilizes hesperidin as well as other water-insoluble flavonoid glycosides. The nature of this material is of considerable interest since it does not seem to be a surfactant type (micelle-forming) solubilizer. To our knowledge, no other types of solubilizing agents have been isolated from plant sources. Accordingly, we have attempted to isolate and characterize this material. This required an assay for solubilizing activity so that the active material could be followed through fractionation procedures. We have developed an assay in which excess hesperidin in dimethyl sulfoxide solution is added to an aqueous solution of the material to be tested. After 3 days, the precipitated material is separated and the solution is analyzed, by high-pressure liquid chromatography (HPLC) on a reversed-phase column, for hesperidin and the two other water-insoluble flavonoids found in lemon leaves, rutin and diosmin. Naringenin is used as an internal standard in the assay. The amounts of each flavonoid in excess of its water solubility are added together to give the total solubilizing activity of the material.

Since the solubilizing factor is of low molecular weight, the initial extraction of the leaves was done with 80% acetone, in which macromolecular material is insoluble. After evaporation of the acetone, water-insoluble material was removed and the aqueous solution was extracted successively with ethyl acetate and butanol. The two extracts and the remaining aqueous phase all contained considerable amounts of activity, which suggests that the leaves may contain more than one solubilizing factor. The concentration of active material was quite low in these fractions, each of which weighed several grams. Therefore, a necessary first step was a large-scale method for removing much of the inactive material. Several column chromatographic procedures (affinity, silica gel, PVP, Sephadex) were tried, but none of them concentrated the active material sufficiently. Success was finally achieved with a reversed-phase adsorbent column. When the aqueous fraction was passed through this column, followed by elution with water, the active material was retained while much

*Work supported in part by the Citrus Products Technical Committee.

of the inactive material was eluted. Fractions enriched in the solubilizing factor were then eluted with increasing concentrations of methanol in water. For final purification of these fractions, a semipreparative HPLC column will be used.

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RECENT STUDIES ON BIOREGULATORS AND CITRUS FRUITS

H. Yokoyama, S. Hasegawa, C. De Benedict,
W-J. Hsu, S. Gold, S. Poling, and E. Hayman
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Fundamental leads, resulting from studies on the mode of action of bioregulators, particularly at the subcellular levels, strongly implicate bioregulatory action in the basic biological processes of the green plant. This action, in large part, helps to explain the multifunctional nature of our bioregulators. So it is not too surprising to observe a number of biochemical and physiological responses on application of the compounds to the green plant. However, these responses are highly concentration-dependent.

These studies profoundly altered our approach to work on plant bioregulation. We now take into consideration the whole plant with the leaf or chlorophyllous tissue being the focal point and the functional nature of the bioregulator or combination of bioregulators used. In this approach, not only the regulation of biochemical and physiological events but also morphological and anatomical characteristics of the plant are considered from its initial stages of development.

Thus, in our work on Citrus, we now attempt to control the development of the fruit from the time just prior to full bloom. The initial application is made at very low concentrations (50-100 ppm); and as the fruit matures, two subsequent applications are made at higher levels (250-500 ppm). The exact level of concentration is determined by the particular bioregulator or combination of bioregulators used.

Preliminary studies, which were conducted in the past season, strongly suggest that the bioregulators affect the maturation process of citrus fruits. On early treatment of navel and Valencia oranges with low concentrations of bioregulators, accelerated maturation and increases in the size of fruits were observed in addition to color enhancement. It is becoming apparent that the bioregulatory action is not limited solely to color enhancement. Particularly with early treatment at low concentrations, other biological responses are observed. These studies will be described and discussed.

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AN OVERVIEW OF CITRUS RESEARCH AT THE
U.S. HORTICULTURAL RESEARCH LABORATORY, ORLANDO, FLORIDA

Roger H. Young*
U.S. Horticultural Research Laboratory
Orlando, Florida

Research challenges for the Florida citrus industry are many and exciting. Our geneticists are currently developing new orange, grapefruit, and tangerine hybrids which have better quality, more acceptability to the consumer, and can withstand freezes better. The 'Sunburst' tangerine hybrid, recently released in Florida, is of superior quality, the tree is cold hardy, and this variety may well replace the 'Dancy' tangerine which matures in November and December. Seedless selections of 'Pineapple' orange and 'Foster' grapefruit have been developed by budwood irradiation. These selections are being field evaluated, and currently the trees appear identical to the original budwood-source trees, except for the seedless character of the fruit. A new family of sweet orange hybrids has been developed whose fruit closely resemble sweet oranges even though one-fourth of the genetic background is Poncirus trifoliata. This is an accomplishment in citrus breeding not before attained. These trees appear to be more cold hardy than sweet orange trees.

Major pathological diseases, namely tristeza virus, foot-rot fungus, nematodes, and blight, as well as the potential threat of a freeze, have required intensive efforts by our rootstock breeders, pathologists, nematologist, and physiologists. Rootstock breeders have made extensive crosses for disease and nematode resistance by using new combinations of parental material as well as Eremocitrus and Microcitrus, which have not been used previously for citrus breeding. New selections are beginning to emerge as candidate rootstocks with improved disease and nematode resistance and cold hardiness. Tristeza virus detection techniques have been greatly improved by an "enzyme-linked immunosorbent assay" (ELISA) and an inclusion body staining procedure. Cross-protection, using a mild tristeza strain as a challenge against severe strains, is being explored as a means to offset the disastrous effects of severe tristeza strains. Research is also underway to determine the basic biochemical mechanisms for cold hardening and nematode resistance. Ridomil, a systemic fungicide, has been cleared for use on nonbearing Florida citrus for phytophthora foot-rot control. The chemical is effective on seedling liners planted in a nursery or on young budded trees as

*Laboratory Director, U.S. Horticultural Research Laboratory, Orlando, and Research Leader, Horticulture-Breeding Unit.

grove replants. Blight, a wiltlike disease, is a serious problem, but the cause has not been determined. To date, the only organism found associated is Fusarium solani. Although Fusarium causes vascular occlusions in seedlings similar to those found in older blighted trees, cause and effect have not been established. Currently, physiologists are investigating the two major characteristics of blight-xylem vessel dysfunction and zinc nutrition imbalance - as both are specific for this disease.

Growth regulators are an integral part of our research on fruit abscission and coloring, tree cold hardiness, and extending the shelf life of fruit by preharvest applications. Several effective chemicals have been developed for fruit abscission. However, with the late-maturing 'Valencia' orange, these abscission chemicals are not nearly as effective in the spring when the trees are in a spring flush. Endogenous growth regulator levels, particularly GA, ABA, IAA, cytokinins, and ethylene and cellulase, are being investigated in relation to the lack of abscission chemical effectiveness in the spring. Ethrel as a preharvest coloring agent was evaluated and is available commercially for tangerines. Bioregulators developed by Henry Yokoyama here at Pasadena are also being evaluated under Florida conditions as potential coloring agents. No chemical has been found yet, however, that increases citrus tree cold hardiness.

Two major thrusts involving insect control have been on citrus blackfly and the sugarcane rootstalk borer weevil. Recently, our laboratory was instrumental in introducing parasites which have brought the citrus blackfly under control. We are also initiating new research on insect vectoring of citrus tristeza virus.

Fruit losses in storage and transit continue to be large, and research is underway to develop better postharvest disease control and storage, handling, packaging, and transit systems. Several new containers have been designed which reduce the amount of misshapened and bruised citrus fruit during transit; and in combination with several recently developed fungicides, decay losses have been reduced significantly. A fruit-chilling treatment is being investigated as an alternative for EDB to control the Caribbean fruit fly larvae prior to shipment.

RECENT WORK ON DIHYDROCHALCONES []

R. M. Horowitz, Bruno Gentili and Stephen M. Poling
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Work has continued during the past year on the identification of metabolites of neohesperidin dihydrochalcone, the synthesis of analogs of this [sweetener], and the structure determination of dihydrochalcones that occur in kumquats.

When neohesperidin dihydrochalcone labeled with carbon-14 on the methylene group β to the keto group is given to rats maintained on a "regular" diet, labeled m-hydroxycinnamic acid and m-hydroxyphenylpropionic acid are found in the urine together with at least two highly polar compounds. The latter compounds appear to be the major metabolites. When the labeled neohesperidin dihydrochalcone is given to rats maintained on a "purified" diet (i.e., one containing no phenolic compounds), isoferulic acid can be detected together with a large number of unidentified compounds. (Rats on the purified diet appear to produce a more complicated series of metabolites than do those on the regular diet.)

During the past year we have synthesized several B-ring analogs of neohesperidin dihydrochalcone. Structure-activity relations of these new ester and C-alkyl derivatives will be discussed. Also to be discussed are some NMR studies which have provided additional information about the structures of the interesting dihydrochalcones obtained from kumquats.

DEBITTERING OF GRAPEFRUIT JUICE WITH IMMOBILIZED NARINGINASE

Alfred C. Olson and Gregory M. Gray
Western Regional Research Center
Berkeley, California 94710

Our earlier finding showed that it was possible to reduce the naringin content of grapefruit juice by using naringinase immobilized in a hollow fiber reactor. While the concept of controlling naringin bitterness with naringinase was proposed over 20 years ago, the use of an immobilized naringinase for this purpose is a relatively new idea. There are several advantages in using immobilized rather than soluble naringinase to debitter grapefruit juice. These include: 1) reuse of the enzyme (less enzyme is required); 2) very accurate control of the level to which one can reduce the naringin; 3) the elimination of any need for further heat inactivation after enzyme treatment; and 4) the absence of particle and cloud loss due to pectin and pulp degradation during enzyme treatment, a potential problem with soluble enzyme methods.

We have recently demonstrated that naringinase obtained from Aspergillus niger and restrained in soluble form in the shell region of a hollow fiber cartridge (Romicon) can be used to reduce very high levels of naringin in grapefruit juice. Single strength unclarified juice from frozen concentrate (obtained from Sunkist Growers, Inc., Ontario, CA) was recycled for different periods of time through a hollow fiber reactor at temperatures up to 45°C. The final level of naringin in the juice was determined by how long the juice was recycled. A sample of original juice containing 885 µg/mL naringin was unacceptable whereas enzyme treated juice containing 225 µg/mL naringin was deemed acceptable by a sensory evaluation panel sensitive to bitterness. The 4 ppm limonin found in the original juice was unchanged by the debittering process. Processing caused no observable changes in juice cloud or suspended particles.

Flow rates high enough to cause turbulent flow in the hollow fiber reactors are recommended in order to minimize concentration polarization and membrane fouling. Recycle flow rates of 10 L/min for the naringinase reactor were studied using a Romicon HFXS unit. At these higher flow rates, demonstrated enzyme activity increased by 75% over recycle rates of 1 L/min in a laboratory unit. Reynolds numbers for 1 L/min and 10 L/min were 290 and 2900 respectively, indicating that at the lower flow rate juice was passing through the fibers in a laminar manner, while at the higher flow rate turbulent flow conditions prevailed. Under

these conditions, the rate of diffusion of substrate (naringin) across the fiber membrane walls was increased, which resulted in more rapid contact between enzyme and substrate and the observed increase in enzyme activity.

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UPDATE ON CITRUS RESEARCH IN TEXAS

Harold E. Brown, Robert R. Cruse and Roger F. Albach
Food Crops Utilization Research Laboratory
Weslaco, Texas

USTA

Ray and Henderson grapefruit are dark-red-fleshed varieties of the Ruby Red grapefruit discovered as "bud-sports" in groves near Mission and Edinburg, Texas. Compositional studies and evaluations of the processing characteristics indicate that the two varieties may be used interchangeably in processing; however, it is recognized that production from present plantings will be directed towards the fresh fruit market. The Henderson has lower Brix and acid, but with a ratio of the same order as the Ray, Ruby Red, and Star Ruby. The lycopene, in particular, decreases as the season advances, accounting for the major color fading in late-season fruit. This is more pronounced in the Ruby Red than in the newer varieties.

Work on processing characteristics of the Star Ruby grapefruit showed the juice to contain 8.8-9.7° Brix, 1.05-1.45% acid, and a lower Brix/acid ratio than the Ruby Red fruit. Vitamin C (24-36 mg/100 ml juice) and naringin (90-645 ppm) were in about the same range in any given season, although some irregular variance occurred. Representatives of the Texas citrus processing industry have indicated that a blend of Star Ruby and Ruby Red juices, containing 20-30% of Star juice at the start of the season and increasing to 40% in the late season, should give acceptable color to commercially processed pink grapefruit juices. The flavor of the juices and blends remains generally acceptable on the average over a 1-year period; however, organoleptic evaluation indicates a shorter period (up to 6 mos) to be preferable for single-strength products. The Star Ruby fruit was noted, both in removal of the pulp from the whole fruit, and in extracting the juice, to have coarser, thicker-walled juice sacs than the Ruby Red.

A study was conducted to determine the effect of moisture on the composition of Marrs and Valencia oranges and Ruby Red grapefruit. Brix, acid, pH, Brix/acid ratio and suspended solids levels were negatively correlated ($P=.01-.10$) with total moisture received when irrigated at various levels of moisture depletion. Naringin in grapefruit showed highest levels at lower moisture conditions. Ascorbic acid (Vitamin C) generally showed no correlation with total moisture; however, this factor is more dependent upon sunlight, rather than moisture. In a second study, emphasizing phases of citrus entomology, and using Marrs, Hamlin, Pineapple, and Valencia oranges, and irrigated only when water stress appeared, negative correlation of Brix, acid, pH, and Brix/acid ratio was most pronounced in the Pineapple variety, less so in the Hamlin and very little in the Marrs. Valencias

correlated somewhat differently, due to different maturity times.

Studies on canned, single strength grapefruit juice indicate that the red color fades upon storage at ambient temperatures (15-36°C). These studies are incomplete, however, there is an indication that the color fades in the can in a similar fashion as on the tree, albeit it is at a slower rate. Work has been initiated to evaluate the cause of color deterioration following storage.

The bitter components, naringin (in grapefruit) and limonin (in oranges and grapefruit), were quantitated along with pulp, pH, acid, °Brix, oil, vitamin C (ascorbic acid) and color in single-strength and reconstituted concentrated juices packed by the major Texas commercial processors over three seasons. By December, for orange, and March, for grapefruit, no juice contained more than 6 ppm limonin. The 3-year mean for oranges was 3.3 ppm limonin and for grapefruit 7.2 ppm limonin and 585 ppm naringin (by the Davis test). In both juices, limonin concentration decreased rapidly as the season progressed; naringin concentration remained steady until spring when it began to increase. The generally perceived decrease in grapefruit juice bitterness during the season is most likely due to loss of limonin and not so much due to naringin decrease. There was found to be no linear correlation between pulp content of either juice and the concentration of the bitter components. Pulp content of orange juice varied considerably and consistently between plants. The 3-year means of vitamin C in grapefruit and orange juices were 31.3 and 43.8 mg/100 ml, respectively.

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NAPHTHAZARIN TOXINS PRODUCED BY FUSARIUM SOLANI: IDENTIFICATION,
OCCURRENCE AND TOXICITY []

J. H. Tatum and R. A. Baker
U.S. Citrus and Subtropical Products Laboratory
Winter Haven, Florida

Fusarium solani isolates were obtained from the roots of field-grown citrus that had blight symptoms and from other sources. These isolates were used to produce both shake and still culture extracts. We have identified nine naphthazarins and one aza anthracenedione from these extracts. Three of these compounds are being reported as metabolites of Fusarium solani for the first time. We are the first to report these compounds as metabolites of Fusarium isolated from Citrus. Toxin formation was dependent on fungal strain, culture medium and age of culture. Some toxins formed in all cultures while others were only formed under specific conditions. Toxicities of the identified naphthazarins will be discussed.

The following compounds were identified: fusarubin; methyl-fusarubin; anhydrofusarubin; javanicin; marticin; isomarticin; rel-(3R,4aR,10aR)- and rel-(3R,4aR,10aS)-5-dioxo-3,4,4a,5,10,10a-hexahydro-7-methoxy-3-methyl-3,6,9-trihydroxy-1H-naphtho[2,3-C]pyran; rel-(3R,4aR,10aR)-5,10-dioxo-3,4,4a,5,10,10a-hexahydro-3,7-dimethoxy-3-methyl,6,9-dihydroxy-1-H-naphtho[2,3-C]pyran; and 5,8-dihydroxy-6-methoxy-3-methyl-2-aza-9,10-anthracenedione.

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CURRENT STATUS OF LIMONOATE DEHYDROGENASE FROM BACTERIA* [1 7

Shin Hasegawa, Carl P. Verdon and Martin F. Schroeder**
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Limonoate dehydrogenases, which catalyze the conversion of limonoate to 17-dehydrolimonoate, have been isolated from three species of bacteria, Arthrobacter globiformis, Pseudomonas NRRL B-5777R and No. 342-152-1. Each dehydrogenase has different characteristics. The dehydrogenases of A. globiformis and No. 342-152-1 require NAD as a cofactor whereas the Pseudomonas dehydrogenase takes NADP preferentially over NAD. Each enzyme has its optimal activity at different pHs and different affinity toward ion exchange and affinity resins. The dehydrogenase of A. globiformis can be isolated very effectively on a small column packed with 4.5 ml of AH-Sepharose 4B-isoobacunoate, which is prepared by attaching the carboxyl group at C-3 of isoobacunoic acid to the amino group of AH-Sepharose 4B, which has a six carbon bridge. The dehydrogenase of Pseudomonas, however, does not have affinity toward the resin under the same conditions.

Limonoate dehydrogenase appears to be an element of an enzyme regulator system that controls the metabolism of limonoids in bacteria. The dehydrogenase of No. 342-152-1 and A. globiformis form a stable enzyme-substrate complex. This complex is so stable that common enzyme preparative procedures, such as sonication, $(\text{NH}_4)_2\text{SO}_4$ precipitation, dialysis and freeze-drying, and ion exchange and affinity column chromatography, do not separate it into two free components. The addition of NAD to the system, however, releases the substrate as a reaction product, 17-dehydrolimonoate. Since the stable complex has affinity toward AH-Sepharose 4B-isoobacunoate similar to the free form, it appears that the catalytic site of the enzyme is not directly involved in the site of the complex formation.

The dehydrogenase of Pseudomonas does not form the stable enzyme-substrate complex. The enzyme has a Hill number of 0.98 with respect to limonoate, indicating that the enzyme exhibits no cooperativity among the subunits; however, there is an inhibitor of the enzyme in cell-free extracts. This inhibitor is present in the extract in an inactive form, which is activated by $(\text{NH}_4)_2\text{SO}_4$ precipitation. This protein-like inhibitor can be separated from the enzyme on a DEAE Sephacel column under conditions in which the column is eluted with increasing salt concentration at pH 7.0. However, the enzyme and inhibitor are eluted

*Work supported in part by the Citrus Products Technical Committee.

**Visiting scientist, Sunkist Growers, Inc., Products Research and Development Division, Ontario, CA.

in the same fractions when the column is eluted at pH 8.0.

The possible use of limonoate dehydrogenase for analyses of limonin in citrus juice also will be discussed. The dehydrogenase of Pseudomonas appears to be a suitable enzyme for this purpose. The enzyme has a K_{mapp} of 0.28 mM and 0.026 mM with respect to limonoate and NADP, respectively, and it is very stable in 2 M $(NH_4)_2SO_4$ solution. The dehydrogenase of A. globiformis, however, is not suitable because of the presence of the stable enzyme-substrate complex, which interferes with the assay.

Pseudomonas cells immobilized in acrylamide gel metabolize limonin in citrus juice. The preliminary test shows that the immobilized cells packed in a column allow substantial reduction of limonin bitterness in navel orange juice.

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CHEMICAL COMPOSITION OF CALIFORNIA
EARLY-, MID-, AND LATE-SEASON NAVEL
ORANGE JUICE, CONCENTRATE, AND PULPWASH*

Carl E. Vandercook, José L. Navarro** and Dora C. Smolensky
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

and

Gary L. Park, Judith L. Beyers, Jo Ann Bradley and Catherine M. Pritz
Sunkist Growers, Inc., Products Research and Development Division,
Ontario, CA

Continued interest in detecting the adulteration of orange juice with sugar, water, or excess (unauthorized) orange pulp wash solids prompted us to begin a regional study of the composition of orange juice and pulp wash. The first stage of the study involved single-strength orange juice, concentrate, and pulp wash solids (two-stage process) from early-, mid-, and late-season California Navel oranges. Samples were analyzed by 42 chemical, physical, and microbiological tests. The study includes simple standard tests (Brix, acidity, formol index, and ash) as well as more specific analyses (individual amino acids and sugars by liquid chromatographic methods, individual organic acids by enzyme assays, vitamin assays). Less specific spectrophotometric analyses and microbiological assays were performed to measure characteristic groups of juice constituents.

The results will be discussed in terms of the similarities, differences and correlations of the parameters to season and/or products. Predictably, the products were compositionally similar, yet there were statistically significant differences between many of the components due to product type and advancing season. An analysis of variance of the data showed a few parameters, such as isocitric acid, were not significantly affected by season or product type. Most parameters (acids, vitamins, spectral properties) showed highly significant effects due to the season. Parameters related to the phenolic compounds were significantly affected by the pulp wash process.

Adulteration of orange juice can be divided into two general classes, i.e., addition of "food ingredients" (water, sugar, acids, etc.) and addition of excess or unauthorized citrus by-products such as pulp wash. The legalities of pulp wash addition and the methods of manufacture vary from one region to another. The pulp wash in this study was made by a two-stage process. Much

*Work supported in part by the Citrus Products Technical Committee.

**Visiting scientist from the Institute de Agroquímica y Tecnología de Alimentos, Valencia, Spain.

of the commercially available pulpwash is made by a multiple stage process, which could result in a product with a significantly different composition. Pulpwash as a product needs to be carefully defined in terms of the manufacturing process, composition, and/or quality relative to the juice.

Differences due to both season and product are observed. Depending on the parameter and type of adulteration, these differences can either increase or decrease the ability to detect adulteration. For example, parameters with large differences due to product are desirable to detect added pulpwash, whereas parameters with small or no differences are better for detecting added sugar and water. Another variation, although not considered in this study, is that due to variety and growing region. Our previous work on total amino acids, for example, showed significant differences between regions (frozen concentrated orange juice: California 3.53; Florida 1.98 meq/100 ml). Both types of differences demonstrate the need to establish and maintain a substantial data base on varietal citrus products from regions around the world.

Given a wide enough data bank, selected parameters and statistical treatments may allow detection of adulteration. In reality, the chosen approach to adulteration detection will be a compromise between analytical costs and acceptable risks. Using the data from this study, several approaches will be demonstrated using chi-square and discriminant analyses with parameters of low variability and low correlation. Multiple regression will be used to demonstrate the use of constituents with a medium to high variability and high correlation.

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Fruit and Vegetable Chemistry Laboratory
263 South Chester Avenue, Pasadena, California 91106

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ISOLIMONIC ACID, A NEW CITRUS LIMONOID

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Shin Hasegawa and Vincent P. Maier
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*Reprints are available at the addresses indicated; patents are available only by purchase at 50¢ a copy from the U.S. Patent Office, Washington, D. C. 29231.

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METHODS FOR THE DETECTION OF ADULTERATION IN PROCESSED CITRUS PRODUCTS

D. R. Petrus and C. E. Vandercook
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Steven Nagy and John A. Attaway, Eds., ACS Symposium Series,
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SOUTHERN REGION

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